



## Materials & Coatings

### Polyimide Aerogel Thin Films

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Polyimide aerogels have been cross-linked through multifunctional amines. This invention builds on "Polyimide Aerogels With Three-Dimensional Cross-Linked Structure," (LEW-18486-1), *NASA Tech Briefs*, Vol. 34, No. 8 (August 2010), page 38, and may be considered as a continuation of that invention, which results in a polyimide aerogel with a flexible, formable form.

Gels formed from polyamic acid solutions, end-capped with anhydrides, and

cross-linked with the multifunctional amines, are chemically imidized and dried using supercritical CO<sub>2</sub> extraction to give aerogels having density around 0.1 to 0.3 g/cm<sup>3</sup>. The aerogels are 80 to 95% porous, and have high surface areas (200 to 600 m<sup>2</sup>/g) and low thermal conductivity (as low as 14 mW/m-K at room temperature). Notably, the cross-linked polyimide aerogels have higher modulus than polymer-reinforced silica aerogels of similar density,

and can be fabricated as both monoliths and thin films.

*This work was done by Mary Ann Meador of Glenn Research Center and Haiquan Guo of Ohio Aerospace Institute. Further information is contained in a TSP (see page 1).*

*Inquiries concerning rights for the commercial use of this invention should be addressed to NASA Glenn Research Center, Innovative Partnerships Office, Attn: Steven Fedor, Mail Stop 4-8, 21000 Brookpark Road, Cleveland, Ohio 44135. Refer to LEW-18864-1.*

### Nanoengineered Thermal Materials Based on Carbon Nanotube Array Composites

**Thermal conductors for integrated circuits and devices can be made from carbon nanotube arrays.**

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State-of-the-art integrated circuits (ICs) for microprocessors routinely dissipate power densities on the order of 50 W/cm<sup>2</sup>. This large power is due to the localized heating of ICs operating at high frequencies and must be managed for future high-frequency microelectronic applications. As the size of components and devices for ICs and other appliances becomes smaller, it becomes more difficult to provide heat dissipation and transport for such components and devices. A thermal conductor for a macro-sized thermal conductor is generally inadequate for use with a micro-sized component or device, in part due to scaling problems.

The cooling of an object by attaching it to a cold reservoir is normally limited by the heat transfer rate across the interface. Except for objects with atomically flat surfaces, practical objects normally have only a very small portion of surface in contact with other solid surfaces. Eutectic bonding materials or thermal conducting pastes/films are normally applied at the interface to increase the contact area. However, the thermal conductivities of these eutectic bonding materials are normally an order of magnitude lower than

those of solid materials such as Cu and Si. The interface thus remains the bottleneck for heat dissipation.

A method has been developed for providing for thermal conduction using an array of carbon nanotubes (CNTs). An array of vertically oriented CNTs is grown on a substrate having high thermal conductivity, and interstitial regions between adjacent CNTs in the array are partly or wholly filled with a filler material having a high thermal conductivity so that at least one end of each CNT is exposed. The exposed end of each CNT is pressed against a surface of an object from which heat is to be removed. The CNT-filler composite adjacent to the substrate provides improved mechanical strength to anchor CNTs in place, and also serves as a heat spreader to improve diffusion of heat flux from the smaller volume (CNTs) to a larger heat sink.

The invention uses an embedded carbon nanotube array to provide one or more high-performance thermal conductors for applications that require large heat dissipation. This approach also improves the mechanical strength of carbon nanotubes (CNTs) so that the CNT array can remain stable and make

good contact to the surface of objects that generate a large amount of heat, through use of reversible buckling and bending of exposed portions of the CNTs. The extremely high thermal conductivity along a carbon nanotube axis is employed to transfer heat away from hot spots in a component or device. Copper and other high-thermal-conductivity materials are deposited to fill interstitial regions or gaps in the first part of a CNT array.

The fabrication involves four steps: (1) Substantially vertically aligned CNT arrays with a preferred length of from 1 to 50 microns are grown on a solid substrate (serving as a heat sink) that has good thermal conductivity, such as Si wafers and metal blocks/films; (2) a first portion of, or all of, interstitial spaces between adjacent CNTs are filled with high-thermal-conductivity materials such as Cu, Ag, Au, Pt, or doped Si by chemical vapor deposition (CVD), physical vapor deposition (PVD), plasma deposition, ion sputtering, electrochemical deposition, or casting from liquid phase; (3) filler materials are removed from a second portion of the interstitial spaces by mechanical polishing (MP),

chemical mechanical polishing (CMP), wet chemical etching, electrochemical etching, or dry plasma etching so that the top portion of the CNT array is exposed, with the bottom part remaining embedded in the filler materials; and (4) the embedded CNT array is applied against an object that is to be cooled. CNTs can reversibly buckle or bend one-by-one under low loading pressure so that a CNT can make maximum contact

with the object to be cooled, even an object with a very rough surface.

Heat can be effectively transferred from the contacting spots along the tube axis to the filler materials as well as the substrates. The filler materials play two critical roles: improving the mechanical stability and maximizing the thermal conductivity. Choosing highly thermal conductive materials as the filler matrix maximizes the heat transfer from the contact spots to the

substrate (i.e., the heat sink or cooling reservoir). An embedded CNT array can be reused without damage or compromise of its heat transport characteristics, in contrast to an approach that relies upon eutectic bonding.

*This work was done by Jun Li and Meyya Meyyappan of Ames Research Center, and Carlos Dangelo of QSolis Inc. Further information is contained in a TSP (see page 1). ARC-15173-1*

## Composite Laminate With Coefficient of Thermal Expansion Matching D263 Glass

**The laminate is a combination of carbon fiber with fiberglass.**

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The International X-ray Observatory project seeks to make an X-ray telescope assembly with 14,000 flexible glass segments. The glass used is commercially available SCHOTT D263 glass. Thermal expansion causes the mirror to distort out of alignment. A housing material is needed that has a matching coefficient of thermal expansion (CTE) so that when temperatures change in the X-ray mirror assembly, the glass and housing pieces expand equally, thus reducing or eliminating distortion. Desirable characteristics of this material include a high stiffness/weight ratio, and low density.

Some metal alloys show promise in matching the CTE of D263 glass, but their density is high compared to aluminum, and their stiffness/weight ratio is not favorable. A laminate made from carbon fiber reinforced plastic (CFRP) should provide more favorable

characteristics, but there has not been any made with the CTE matching D263 Glass.

It is common to create CFRP laminates of various CTEs by stacking layers of "prepreg" material at various angles. However, the CTE of D263 glass is 6.3 ppm/°C at 20 °C, which is quite high, and actually unachievable solely with carbon fiber and resin.

A composite laminate has been developed that has a coefficient of thermal expansion identical to that of SCHOTT D263 glass. The laminate is made of a combination of T300 carbon fiber, E-glass, and RS3C resin. The laminate has 50% uni-T300 plies and 50% uni-E-glass plies, with each fiber-layer type laid up in a quasi-isotropic laminate for a total of 16 plies. The fiber volume (percent of fiber compared to the resin) controls the CTE to a great extent. Tests have con-

firmed that a fiber volume around 48% gives a CTE of 6.3 ppm/°C. This is a fairly simple composite laminate, following well established industry procedures.

The unique feature of this laminate is a somewhat unusual combination of carbon fiber with E-glass (fiberglass). The advantage is that the resulting CTE comes out to 6.3 ppm/°C at 20 °C, which matches D263 glass. The trick with this laminate is to establish the proper fiber volume to get the desired CTE. Laminates were made with several different fiber volumes and coupons were tested to establish the relationship between fiber volume and CTE. Testing proved that fiber volume should be about 48%.

*This work was done by David Robinson and Benjamin Rodini of Goddard Space Flight Center. Further information is contained in a TSP (see page 1). GSC-16261-1*